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(56) Documents cited
GB 2223255 A EP 0386636 A EP 0382071 A
EP 0374671 A

(58) Field of search
UK CL (Edition K) B1V VE, E1F FGP
INT CL$^5$ C09K

# (54) Invert emulsion for use in a drilling fluid

(57) An invert emulsion, low toxicity drilling fluid comprising esters and alcohols is disclosed. The esters may be derived from vegetable oil, fatty acids and the alcohols may be ethanol, isopropanol, ethylene glycol or glycerol. This fluid has a low water activity and thus minimizes destabilization of the borehole.

GB 2 251 447 A

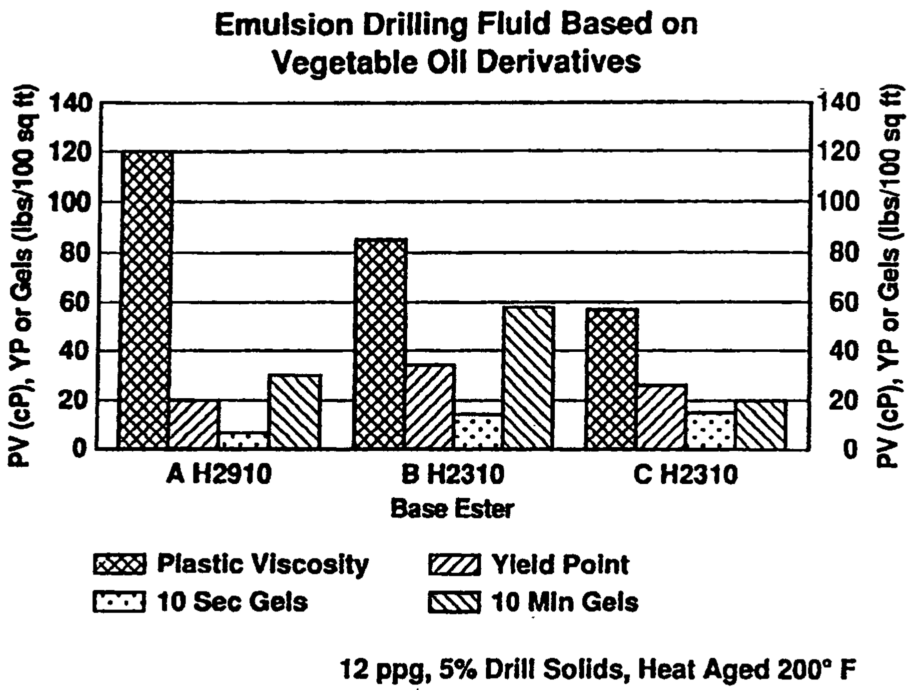
Emulsion Drilling Fluid Based on
Vegetable Oil Derivatives
PV (cP), YP or Gels (lbs/100 sq ft)
0
20
40
60
80
100
120
140
A H2910
B H2310
C H2310
Base Ester
Plastic Viscosity
Yield Point
10 Sec Gels
10 Min Gels
12 ppg, 5% Drill Solids, Heat Aged 200° F

*FIG. 1*

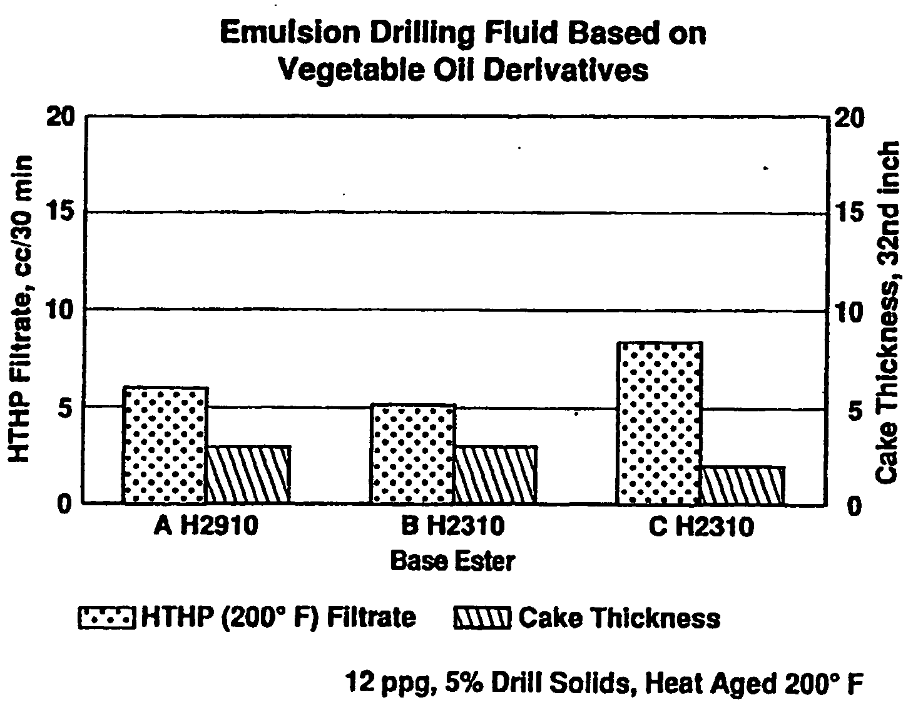
Emulsion Drilling Fluid Based on
Vegetable Oil Derivatives
HTHP Filtrate, cc/30 min
Cake Thickness, 32nd inch
0
5
10
15
20
A H2910
B H2310
C H2310
Base Ester
HTHP (200° F) Filtrate
Cake Thickness
12 ppg, 5% Drill Solids, Heat Aged 200° F

*FIG. 2*

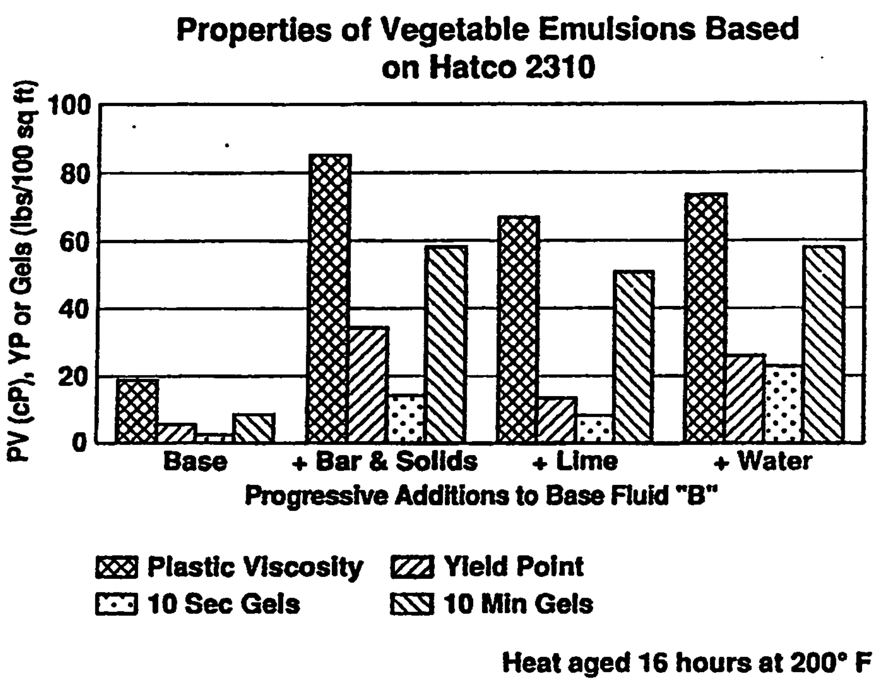
Properties of Vegetable Emulsions Based on Hatco 2310
PV (cP), YP or Gels (lbs/100 sq ft)
100
80
60
40
20
0
Base
+ Bar & Solids
+ Lime
+ Water
Progressive Additions to Base Fluid "B"
Plastic Viscosity
Yield Point
10 Sec Gels
10 Min Gels
Heat aged 16 hours at 200° F

*FIG. 3*

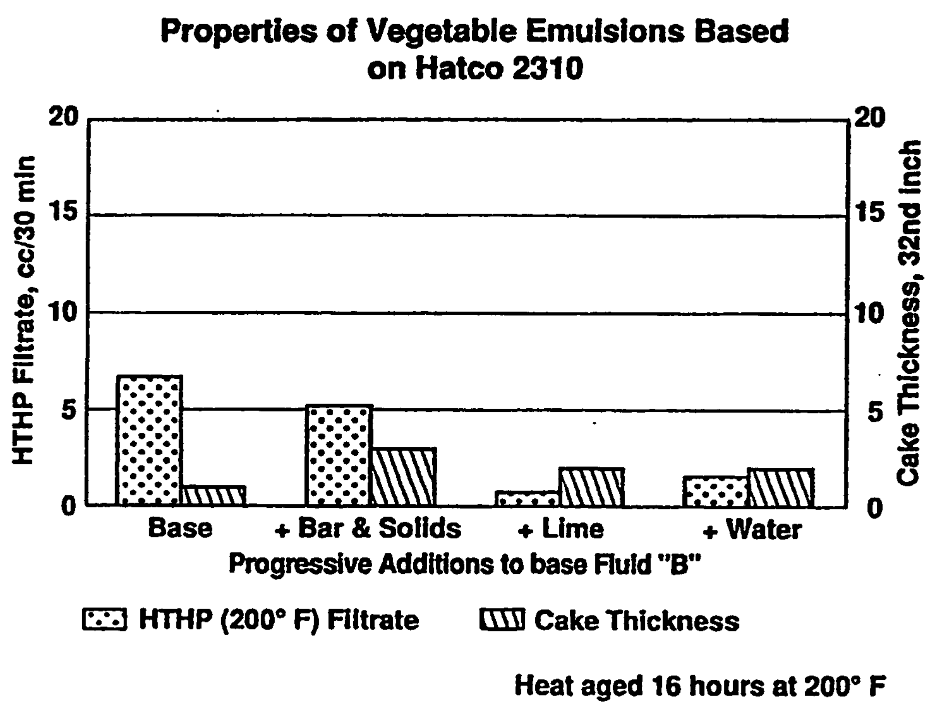
Properties of Vegetable Emulsions Based on Hatco 2310
HTHP Filtrate, cc/30 min
Cake Thickness, 32nd Inch
20
15
10
5
0
Base
+ Bar & Solids
+ Lime
+ Water
Progressive Additions to base Fluid "B"
HTHP (200° F) Filtrate
Cake Thickness
Heat aged 16 hours at 200° F

*FIG. 4*

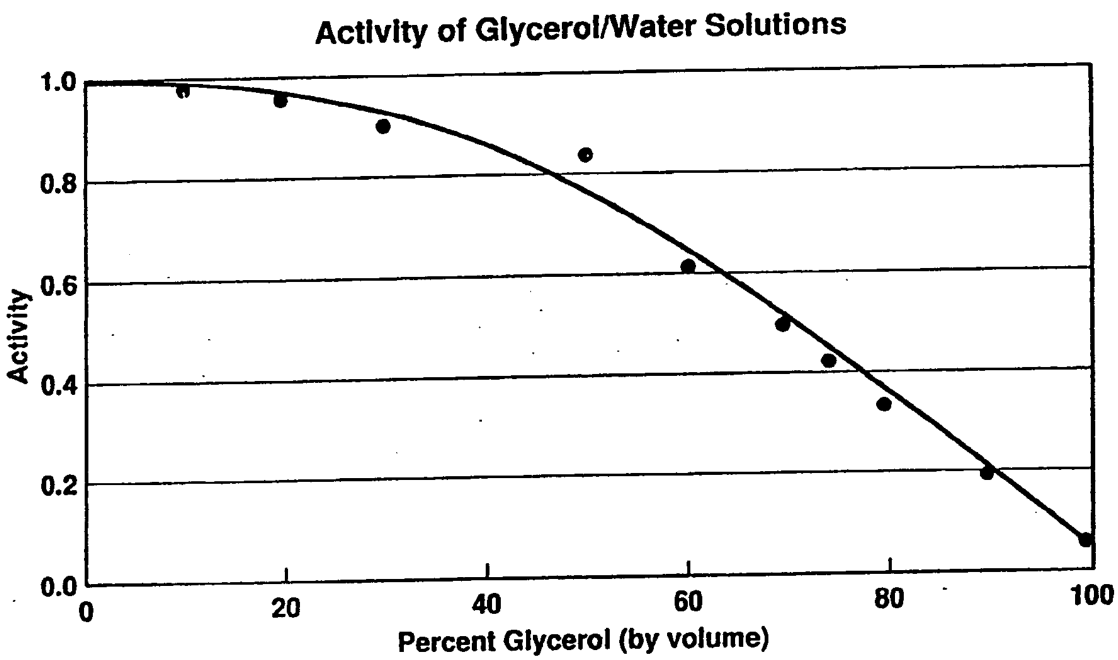
Activity of Glycerol/Water Solutions
1.0
0.8
0.6
0.4
0.2
0.0
Activity
0
20
40
60
80
100
Percent Glycerol (by volume)

*FIG. 5*

INVERT EMULSION FOR USE IN A DRILLING FLUID

This invention pertains to low toxicity invert emulsion drilling fluids, the use of which results in minimal loss of borehole stability.

It is well known in the art that drilling fluids must be used in connection with the drilling of wells, such as those in the oil and gas industry. Such drilling fluids serve several functions in the drilling process. These functions include: removal of drilled cuttings, sealing of the sides of the wellbore so as to minimize drilling fluid loss into the formation, provision of a hydrostatic head to prevent blowouts from high pressure fluids entering into the wellbore and flowing to the surface, creation of a low-friction surface on the wellbore to facilitate rotation and removal of the drill string as operational conditions require, cooling of the drill bit and lubrication to prevent the drill pipe from sticking during rotation.

Drilling fluids have typically been colloidal suspensions of certain viscosifiers and filtration control materials, such as clays, as well as of fine drilled solids, in either oil or water. Various chemicals are added to alter, enhance, influence or modify the properties of the suspension, using techniques which are known to one skilled in the art. For example, a weighting agent, such as barium sulfate, commonly known as "barite," is added to increase the density of the fluid. Gellants are used to increase viscosity and gel strength and provide suspension

properties. Filtration control materials,such as soluble polymers or asphaltic materials, are added to encourage the development of the filter cake on the sides of the wellbore so that a minimal amount of the drilling fluid will enter a permeable formation.

Because of their better thermal stability as compared to water-based fluids, oil-based water-in-oil emulsion fluids have generally been used in high temperature applications. However, as the environmental impact of the disposal of these spent slurries, and the drilled cuttings carried by these slurries, has become increasingly scrutinized, water-based fluids have become more and more the fluid of choice in the industry. Unfortunately, the use of water-based fluids is often undesirable because the water is absorbed by the shales, causing the shales to weaken, and leading ultimately to loss of integrity and failure of the wellbore.

Conventional drilling fluids of the related art, which fluids prevent hydration of the shale formations surrounding the wellbore, are water-in-oil emulsions of diesel oil or mineral oil, together with brine, stabilized by surface active materials. These water-in-oil emulsions are generally known as invert emulsions. The brine internal phase of the emulsion is intended to prevent osmotic transfer of the water from the drilling fluid to the shale by maintaining a chemical activity of the water phase equal to or below that of the water in the shale. This osmotic transfer of water to the shale is what destabilizes the shale, resulting in borehole breakdown. In order to achieve the required chemical activity depression, inorganic salts such as calcium chloride, sodium chloride, or potassium acetate are added and may be required in concentrations of up to about 70% by weight of the aqueous phase. For

example, in United Kingdom Patent Application 2,223,255A by Shell Internationale Research Maatschappij B. V., the invert emulsion drilling fluid disclosed demonstrated improved properties with increase in salt concentration, and thus teaches the necessity of salt in such fluids.

The major disadvantage of these more conventional emulsified drilling fluids is that the petroleum-derived oils which they contain are often toxic to marine organisms, cause skin irritation in humans and animals, and are slow to biodegrade. Furthermore, the inorganic salts required for chemical activity depression of the water phase are toxic to land plants and inhibit biodegradation of the drilling fluid.

The present invention addresses these competing concerns of environmental acceptability of the spent drilling fluid and operational acceptability of the circulating drilling fluid. These concerns are resolved by using an emulsified drilling fluid wherein an ester constitutes the continuous oleaginous phase and a polar organic liquid such as a polyalcohol constitutes the internal dispersed phase.

According to the invention from one aspect there is provided an invert emulsion for use as a component of a drilling fluid, comprising an ester or esters with a viscosity between about 3 cp. ($3 \times 10^{-3}$ Pa s) and about 12 cp. ($12 \times 10^{-3}$ Pa s) at 40°C, said ester or esters being emulsified with a polar organic liquid of the class comprising alcohols, polyalcohols, ethanol, isopropanol, ethylene glycol, and glycerol.

According to the invention from another aspect there is provided a process for making an invert emulsion for use as a component of a

drilling fluid, comprising

a) separation of glycerine from a vegetable oil or vegetable oils, leaving substantially only fatty acids;

b) esterification of said fatty acids to form esters with a viscosity between about 3 cp. ($3 \times 10^{-3}$ Pa s) and about 12 cp. ($12 \times 10^{-3}$ Pa s) at 40°C.

c) recombination of said glycerine with said fatty acid esters.

This invention addresses the problem of providing an invert emulsion drilling fluid which is formulated using predominantly non-toxic or low-toxicity, biodegradable materials. It is an advantage that a drilling fluid produced using the methods disclosed herein can have an extremely low toxicity. It is a further advantage that a drilling fluid produced using the methods disclosed herein can have very low water activities, thus minimizing destabilization of the wellbore. It is a further advantage that a drilling fluid produced using the methods disclosed herein may not need to have any inorganic salts added, without compromising its efficacy.

The drilling fluids disclosed hereinbelow include invert emulsions of polyalcohols, such as glycerol, in esters, which are organic liquids. Therefore, the esters constitute the continuous phase, with the polyalcohols being dispersed in the esters. These drilling fluids may be thought of as an invert emulsion drilling fluid, as described above, wherein the oil phase is replaced by esters and the brine phase is replaced by polyalcohols.

The esters are preferably derivatives of natural vegetable oils such as palm oil and coconut oil, but can also be derived from a variety of vegetable oils and even animal fats. The viscosity of the esters ranges from about 3 cp. ($3 \times 10^{-3}$ Pa s) to about 12 cp. ($12 \times 10^{-3}$ Pa s) at 40°C, and in the preferred embodiment ranges from about 3 cp. ($3 \times 10^{-3}$ Pa s) to about 5 cp. ($5 \times 10^{-3}$ Pa s) at 40°C. The esters provide the lubricity and anticorrosion benefits, while the polyalcohols provide the low chemical activity environment that attracts water that may enter the fluid during the drilling process and prevents it from entering the shale formations. Water contaminants entering the drilling fluid will be incorporated into the polyalcohol phase and will be inhibited from osmotically transferring into the shale formations.

Mere substitution of an ester for the oil phase of conventional emulsified drilling fluids does not satisfy the simultaneous requirements of shale inhibition, low toxicity, and biodegradability. This is because high salt concentrations required in the aqueous phase for chemical activity suppression still render these systems unacceptably toxic to land plants and some marine life. Similarly, incorporation of polyalcohols into emulsions with petroleum based oils does not produce a drilling fluid which satisfies the low toxicity and biodegradability requirements because of the presence of the petroleum oils.

Preferred ways of performing this invention combine substances that were originally separated from similar natural plant or animal products, into a nontoxic, biodegradable, chemically inhibitive drilling fluid. For example, the refining of natural plant or animal fats and oils produces glycerol and fatty acids. Fatty acids can be combined with

various alcohols to produce esters. Glycerol, in turn is emulsified into these esters to produce an invert drilling fluid. Additionally, many surfactants that promote emulsification are also derivatives of fatty acids and glycerol. The intent of this example is not to strictly limit the application to polyalcohols and esters derived from one particular plant or animal precursor, or to limit the polyalcohol, ester, and surfactant derivatives to be entirely derived from plant or animal precursors, but to show that the major components are largely derived from natural, non-hydrocarbon products.

For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, wherein :-

FIGURE 1 compares the rheological properties of three example formulations A, B and C (See Tables 1 to 3 hereinbelow), each prepared with 5 volume percent simulated drill solids. Plastic viscosity (PV) is a measure of the dynamic high shear rate viscous properties of the fluids, while yield point (YP) and gels are a measure of the low shear rate gel properties of fluids necessary for solids suspension. A 10 second gel strength of 4 lbs/100 sq. ft. (1.9 kPa) is considered necessary for solids suspension, while desired yield point may lie in the range of 5 - 35 lbs/100 sq. ft. (2.4-16.8 kPa) depending on flow rate and wellbore angle. FIGURE 1 shows that the example fluids containing a typical amount of drill solids satisfy the solids suspension requirements.

FIGURE 2 compares the high temperature filtration properties of each of the formulations that were prepared with 5 volume percent drill solids. This test was done according to API RP 13B-2, June 1, 1990 edition. Under the conditions of this test, filtrate volumes should be less than 10cc's/30 minutes for an invert emulsion fluid, and filter cake thickness should be less than 3/32nd inch (2.4 mm). This drawing shows that these fluids satisfy these requirements.

FIGURE 3 shows the effect on rheological properties of progressively adding solids and contaminants to one of the particular example formulations. In this case the formulation was based on example formulation "B". The properties of the base fluid are compared with those containing barite + simulated drill solids, barite + drill solids + lime simulated cement contamination, and then all of the foregoing + water contamination.

FIGURE 4 shows the effect of the above progressive contaminant addition on the high temperature filtration properties of example fluid "B".

FIGURE 5 shows the activity depressing effect of glycerol. These data are used infra to illustrate that even if the drilling fluid is contaminated with five volume percent fresh water, the activity of the system will still be sufficiently depressed to prevent hydration of most shales, based upon ten volume percent glycerol in the fluid.

A preferred embodiment of this invention is a formulation that contains an ester concentration of between 60-98 volume percent of total

liquids, and a polyalcohol concentration between 2 and 40 volume percent of total liquids. The esters preferably have a viscosity between about 3 cp. ($3 \times 10^{-3}$ Pa s) and about 5 cp. ($5 \times 10^{-3}$ Pa s) at 40° C.

Additionally, the presence of emulsifiers as described earlier, is necessary to stabilize the emulsion. Fluid loss should be reduced with up to 25 lbs/bbl (71.4 gm/litre) gilsonite or oxidized asphalt, and up to 25 lbs/bbl (71.4 gm/litre) organophilic lignite, each separately or in combination. Gel strength can be augmented with organophilic clay, up to 20 lbs/bbl (57.1 gm/litre). Chemical scavengers, such as zinc oxide ($ZnO$) can be added as necessary to immobilize hydrogen sulfide by producing insoluble zinc sulfide. Use of traditional sulfide scavengers such as $Ca(OH)_2$ (lime) must be minimized to below about 2 lbs/bbl (5.7 gm/litre) in order to prevent extensive hydrolysis of the esters.

## EXAMPLES

In the following examples, the emulsifiers, filtration control additives, and rheology control additives were selected from among those used in conventional water-in-oil invert emulsion drilling fluids. Emulsifiers can include fatty acid soaps, alkanolamides, long chain carboxylic acid esters, glyceryl and polyglyceryl esters of natural fatty acids, polyoxyethylene glycol esters and polyoxyethylenated fatty acids, amines and their salts, and also mixtures of the above and other nonionic and cationic surfactants.

Three drilling fluid formulations, A, B, and C, comprising glycerol/ ester emulsions, were prepared. Their compositions and test results are

summarized in Tables 1, 2, and 3. With each formulation, an unweighted base fluid without simulated contaminants was prepared and tested, as well as fluids containing barite weighting material to give 12 lb/gal (1438 kg/$m^3$) density, simulated drilled solids at 5 vol. %, water simulated contaminant at 5 vol. %, and lime to simulate cement contamination, as indicated in Tables 1, 2, and 3.

The drilling fluid properties, except water activity were measured using standard procedures specified in API Bulletin RP 13B-2, "Recommended Practice Standard Procedure for Field Testing Oil-Based Drilling Fluids," 1st Edition, 6/1/90. Rheology was measured with a concentric rotational viscometer, and filtration properties were measured with Baroid filtration cell. The gel strength notation in the tables refers to readings after 10 second and 10 minute static periods respectively.

Water activity measurements were made using a General Eastern Model 880 Electrohygrometer. An electrohygrometer measures relative humidity above samples of liquids, which is approximately linearly related to water activity in the liquids. A calibration curve was first constructed by placing 40 ml of each of several calibration solutions into a 150 ml flask (Fleaker from Dow Corning) and measuring the relative humidity in the closed air space above each sample after 30 minute equilibration periods. The electrohygrometer probe was inserted through a hole bored through the rubber Fleaker cap in such a way that air did not enter the flask by passing between the probe body and cap. The readings from the electrohygrometer will range between 25-95 units of relative humidity,

and these readings are calibrated against the known activities of the several calibration standards at 25° C:

| Solution | Water Activity |
|---|---|
| Saturated calcium chloride | 0.295 |
| Saturated calcium nitrate | 0.505 |
| Saturated sodium chloride | 0.753 |
| Saturated potassium nitrate | 0.938 |

Having calibrated the instrument, the samples of drilling fluid or glycerol solutions were separately introduced into the Fleaker flask, equilibrated for 30 minutes at 25°C and measured for relative humidity, from which the activities were obtained using the calibration curve.

These drilling fluid formulations were prepared by adding the emulsifiers to the ester under low shear for 10 minutes in a Hamilton Beach Multimixer, then adding the fluid loss materials at low shear for 5 minutes, followed by the polyalcohol under medium shear for 5 minutes. The organophilic clay gellant was then added under medium shear for 15 minutes. When added, weight material, simulated drill solids, lime, and water were introduced after the clay, and the final mixing was done at medium shear for 45 minutes. In the formulations, the scaling factors between these experimental formulations and the simulated real situation are that one gram is equivalent to one lb and 350 ml is equivalent to a barrel, and one gram/350 ml is equivalent to 1 lb/bbl.

Prior to measurement of properties, each of the formulations was heat aged in a metal cylinder under gentle rotation for 16 hours, a process known as hot rolling. The formulations were subjected to this process in an attempt to simulate thermal aging of the drilling fluids, so that their thermal stability could be tested.

In these formulations, the esters 2310 and 2910 were obtained from Hatco Corporation, Witco 3222 and Witco 511 are emulsifiers from Witco Corporation, Truvis is an organophilic clay from International Drilling Fluids, and Emul VR is a copolymer suspending aid from Doverstrand, Ltd. Gilsonite is an asphaltic fluid loss material from American Gilsonite Company.

EXAMPLE 1

Formulation A is based on di-isodecyl adipate ester (Hatco 2910) which has a viscosity of 10 cp. ($10 \times 10^{-3}$ Pa s) at 120 degrees Fahrenheit (48.9°C). As shown in Table 1, the base fluid A had low gel strength and hence poor suspending properties, until weighting material and simulated drill solids were added to produce a fluid, A', with typical drilling fluid composition.

As would be recognized by one skilled in the art, the fluid properties of the system with drill solids and barite (A') are good by accepted standards for drilling fluids, although the plastic viscosity is somewhat high. The gel strength and yield point of fluid A' are suitable for adequate solids suspension. The filtration control is also satisfactory, with a high temperature (200°F or 93.3°C) filtrate of 6cc/30

minutes, and a filter cake 3/32 inch (2.4 mm) thick. Good quality water-based drilling fluids typical of the art exhibit typical high temperature high pressure("HTHP") filtrate values near 20 cc/30 minutes at this temperature. Thus, the formulation of this Example has a much better filtration than the drilling fluids typical of the related art. The rheology and filtration properties of system A' are summarized in Figures 1 and 2.

Lime and water contaminants added to this formulation not only did not render the fluid properties unacceptable from a drilling standpoint, but in fact did not significantly alter the measured properties.

The water activity of the fluid increased to 0.575 upon addition of five percent by volume water (A''', Table 1). This value is still sufficiently low to inhibit water transfer to most shales.

## EXAMPLE 2

This formulation, Series B, was based on Hatco 2310, an iso-decyl ester of C8-C10 monocarboxylic acids. This is a lower viscosity (4 cp. at 120°F or 4 x $10^{-3}$ Pa S at 48.9°C) ester and was chosen to produce a drilling fluid of lower plastic viscosity, which is desirable to minimize required pump outlet pressure for effective circulation. However, to offset the lower gel strength and yield point expected to accompany the lower viscosity ester, the amount of the organophilic clay Truvis was doubled to 10 lbs/bbl (28.6 gm/litre).

This fluid also had rheological properties suitable for a drilling fluid, as shown in Table 2. Plastic viscosities of B' and B", which fluids contained drill solids, and drill solids and lime, respectively, were significantly lower than those of equivalent samples of series A fluids, although gel strengths and yield points were higher. The HTHP filtration volumes were also lower, and filter cake was suitably thin. The properties for the weighted sample contaminated with drill solids (B') appear in Figures 1 and 2. The fluid retained acceptable properties even after progressive addition of contaminants (Figures 3 and 4).

After adding water as a contaminant, activity increased to 0.615, still acceptably low for most drilling applications.

## EXAMPLE 3

Formulation series "C," with properties summarized in Table 3, was also based on Hatco 2310, an iso-decyl ester of C8-C10 monocarboxylic acids. In order to effect further reduction in viscosity, this formulation did not contain Emul VR. An additional surfactant, Witco 511, was added to provide increased stability to the emulsion.

As a consequence of omitting the supplemental gellant, Emul VR, viscosity of this formulation was the lowest of all the series of formulations examined in these examples, although yield point and gel strengths remained within values generally accepted as necessary for suspending solids and removing cuttings from a wellbore (Figure 1). Filtration properties of this formulation were acceptable for a drilling fluid (Figure 2).

Water contamination (C"') did not raise the water activity above 0.379 (Table 3), which is adequate to inhibit hydration of most shales.

## EXAMPLE 4

In this Example, the water activities of glycerol/water solutions were measured. The results of these measurements in Figure 5 show that glycerol is capable of maintaining sufficiently low activities to prevent hydration of shales in the eventuality of moderate brine influxes in the course of drilling. For example, an influx of water of 5 vol. % of the drilling fluid volume would cause a 33% dilution of the glycerol phase [based upon 10% glycerol emulsified in the ester phase], which would increase water activity to about 0.55. As is well known to one skilled in the art, this water activity is below the activities of most shales, and therefore such a drilling fluid would tend not to osmotically transfer water to such shales.

TABLE 1

TEST SERIES A

| COMPONENT | A | A' | A'' | A''' |
|---|---|---|---|---|
| HATCO 2910 ESTER (ml) | 315 | 246 | 246 | 246 |
| GLYCEROL (ml) | 35 | 33 | 33 | 33 |
| WITCO 3222 (grams) | 5 | 5 | 5 | 5 |
| TRUVIS (grams) | 5 | 5 | 5 | 5 |
| GILSONITE (grams) | 10 | 10 | 10 | 10 |
| BW EMUL VR (grams) | 4 | 4 | 4 | 4 |
| DRILL SOLIDS (grams) | ---- | 46 | 46 | 46 |
| BARITE (grams) | ---- | 224 | 224 | 224 |
| LIME (grams) | ---- | ---- | 2 | ---- |
| WATER (ml) | ---- | ---- | ---- | 17 |

| PROPERTY | A | A' | A'' | A''' |
|---|---|---|---|---|
| PLASTIC VISCOSITY (PV) cp (Pa s) | 12 ($12 \times 10^{-3}$) | 120 ($120 \times 10^{-3}$) | 101 ($101 \times 10^{-3}$) | 71 ($71 \times 10^{-3}$) |
| YIELD POINT (YP) lbs/100 sq ft ($gm/m^2$) | 0 (0) | 20 (976) | 8 (391) | 18 (879) |
| GELS lbs/100 sq ft ($gm/m^2$) | 0/3 (0) | 6/30 (9.8) | 2/13 (7.5) | 2/2 (48.8) |
| API FILTRATE, cc's/30 min | 0.3 | 1.1 | 0 | 0 |
| CAKE THICKNESS, inches (mm) | 1/32 (0.8) | 1/32 (0.8) | 1/32 (0.8) | 1/32 (0.8) |
| HTHP FILTRATE cc's/30 min | 6.0 | 6.0 | 6.8 | 1.0 |
| CAKE THICKNESS, inches (mm) | 1/32 (0.8) | 3/32 (2.4) | 2/32 (1.6) | 3/32 (2.4) |
| ACTIVITY | 0.236 | 0.230 | 0.240 | 0.575 |

TABLE 2

TEST SERIES B

| COMPONENT | B | B' | B'' | B''' |
|---|---|---|---|---|
| HATCO 2310 ESTER (ml) | 315 | 246 | 246 | 246 |
| GLYCEROL (ml) | 33 | 33 | 33 | 33 |
| WITCO 3222 (grams) | 5 | 5 | 5 | 5 |
| TRUVIS (grams) | 10 | 10 | 10 | 10 |
| GILSONITE (grams) | 10 | 10 | 10 | 10 |
| BW EMUL VR (grams) | 4 | 4 | 4 | 4 |
| DRILL SOLIDS (grams) | ---- | 46 | 46 | 46 |
| BARITE (grams) | ---- | 224 | 224 | 224 |
| LIME (grams) | ---- | ---- | 2 | 2 |
| WATER (ml) | ---- | ---- | ---- | 17 |

| PROPERTY | B | B' | B'' | B''' |
|---|---|---|---|---|
| PLASTIC VISCOSITY (PV), cp (Pa s) | 19 ($19 \times 10^{-3}$) | 85 ($85 \times 10^{-3}$) | 67 ($67 \times 10^{-3}$) | 73 ($73 \times 10^{-3}$) |
| YIELD POINT (YP), lbs/100 sq ft ($gm/m^2$) | 6 (293) | 34 (1660) | 13 (635) | 26 (1269) |
| GELS, lbs/100 sq ft ($gm/m^2$) | 3/9 (16.2) | 14/58 (11.8) | 8/51 (7.7) | 23/58 (19.4) |
| API FILTRATE, cc's/30 min | 0.5 | 1.1 | 0.1 | 0.1 |
| CAKE THICKNESS, Inches (mm) | 1/32 (0.8) | 2/32 (1.6) | 1/32 (0.8) | 1/32 (0.8) |
| HTHP FILTRATE, cc's/30 min | 6.8 | 5.2 | 0.8 | 1.6 |
| CAKE THICKNESS, Inches (mm) | 1/32 (0.8) | 3/32 (2.4) | 2/32 (1.6) | 2/32 (1.6) |
| ACTIVITY | 0.211 | 0.236 | 0.257 | 0.615 |

TABLE 3
TEST SERIES C

| COMPONENT | C | C' | C'' | C''' |
|---|---|---|---|---|
| 2310 ESTER (ml) | 315 | 246 | 246 | 246 |
| GLYCEROL (ml) | 33 | 33 | 33 | 33 |
| WITCO 3222 (grams) | 5 | 5 | 5 | 5 |
| WITCO 511 (grams) | 5 | 5 | 5 | 5 |
| TRUVIS (grams) | 10 | 10 | 10 | 10 |
| GILSONITE (grams) | 10 | 10 | 10 | 10 |
| DRILL SOLIDS (grams) | ---- | 46 | 46 | 46 |
| BARITE (grams) | ---- | 224 | 224 | 224 |
| LIME (grams) | ---- | --- | 2 | 2 |
| WATER (ml) | ---- | --- | --- | 17 |

| PROPERTY | C | C' | C'' | C''' |
|---|---|---|---|---|
| PLASTIC VISCOSITY (PV), cp (Pa s) | 15 ($15 \times 10^{-3}$) | 57 ($57 \times 10^{-3}$) | 51 ($51 \times 10^{-3}$) | 50 ($50 \times 10^{-3}$) |
| YIELD POINT (YP), lbs/100 sq ft ($gm/m^2$) | 5 (244) | 26 (1269) | 20 (976) | 20 (976) |
| GELS lbs/100 sq ft ($gm/m^2$) | 5/5 (48.9) | 15/20 (36.6) | 14/16 (42.7) | 13/16 (39.7) |
| API FILTRATE, cc's/30 min | 6.0 | 2.8 | 4.0 | 2.1 |
| CAKE THICKNESS, Inches (mm) | 1/32 (0.8) | 2/32 (1.6) | 2/32 (1.6) | 2/32 (1.6) |
| HTHP FILTRATE, cc's/30 min | 10.0 | 8.4 | 12.0 | 10.4 |
| CAKE THICKNESS, Inches (mm) | 1/32 (0.8) | 2/32 (1.6) | 2/32 (1.6) | 2/32 (1.6) |
| ACTIVITY | 0.148 | 0.159 | 0.163 | 0.379 |

CLAIMS:

1. An invert emulsion for use as a component of a drilling fluid component comprising an ester or esters with a viscosity between about 3 cp. ($3 \times 10^{-3}$ Pa s) and about 12 cp. ($12 \times 10^{-3}$ Pa s) at 40°C, said ester or esters being emulsified with a polar organic liquid of the class comprising alcohols, polyalcohols, ethanol, isopropanol, ethylene glycol, and glycerol.

2. An invert emulsion according to claim 1, wherein the polar organic liquid is glycerol.

3. An invert emulsion according to claim 1 or 2, wherein said esters are of fatty acids derived from vegetable oils.

4. An invert emulsion according to any preceding claim, wherein the ester concentration is between 60-98 volume percent of the emulsion and the polar organic liquid concentration is between 2 and 40 volume percent of the emulsion.

5. A process for making an invert emulsion for use as a component of a drilling fluid, comprising

a) separation of glycerine from a vegetable oil or vegetable oils, leaving substantially only fatty acids;

b) esterification of said fatty acids to form esters with a viscosity between about 3 cp. ($3 \times 10^{-3}$ Pa s) and about 12 cp. ($12 \times 10^{-3}$ Pa s) at 40°C); and

c) recombination of said glycerine with said fatty acids esters.

6. An invert emulsion made by a process according to claim 5.

7. A drilling fluid containing an invert emulsion according to any of claim 1, 2, 3, 4, or 6.

8. A method of drilling a well using a drilling fluid according to claim 7.

9. An invert emulsion for use as a component of a drilling fluid, substantially as hereinbefore described with reference to the accompanying drawings.

10. A process for making an invert emulsion for use as a component of a drilling fluid, substantially as hereinbefore described with reference to the accompanying drawings.

| Patents Act 1977<br>Examiner's report to the Comptroller under Section 17 (The Search Report) | Application number<br>9200007.4 |
|---|---|
| **Relevant Technical fields**<br>(i) UK Cl (Edition K ) E1F (FGP);B1V (VE)<br>(ii) Int Cl (Edition 5 ) C09K | Search Examiner<br>D B PEPPER |
| **Databases (see over)**<br>(i) UK Patent Office<br>(ii) | Date of Search<br>5 FEBRUARY 1992 |

Documents considered relevant following a search in respect of claims 1 TO 10

| Category (see over) | Identity of document and relevant passages | Relevant to claim(s) |
|---|---|---|
| Y | GB 2223255 A (SHELL) | 1,2,4 |
| Y | EP 0386636 A (HENKEL) | 1,2,4 |
| Y | EP 0382071 A (HENKEL) | 1,2,4 |
| Y | EP 0374671 A (HENKEL) | 1,2,4 |

| Category | Identity of document and relevant passages | Relevant to claim(s) |
|---|---|---|
| | | |

## Categories of documents

X: Document indicating lack of novelty or of inventive step.

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A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

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